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(54) POWDER COATING COMPOSITION, ITS MANUFACTURING METHOD AND METHOD FOR FORMING COATING FILM USING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a novel powder coating composition which gives an excellent metallic appearance.

SOLUTION: The powder coating composition is obtained by mixing metal flakes having adsorbed a leafing agent on the surface thereof with a thermosetting resin powder or by bonding metal flakes having adsorbed a leafing agent on the surface thereof to the surface of a thermosetting resin powder.

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CLAIMS

[Claim(s)]

[Claim 1] The powder-coatings constituent which comes to mix thermosetting resin fine particles with the metal flake which made the leafing-ized agent stick to a front face.

[Claim 2] The powder-coatings constituent which makes the metal flake to which the leafing-ized agent was made to stick come to adhere to the front face of thermosetting resin fine particles.

[Claim 3] The powder-coatings constituent of claims 1 or 2 whose metal flakes are 1 to 20 weight section to the thermosetting resin fine-particles 100 weight section.

[Claim 4] claims 1-3 whose leafing-ized agents are the orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol -- either -- the powder-coatings constituent of a publication.

[Claim 5] claims 1-3 whose leafing-ized agents are the saturated fatty acid of carbon numbers 12-18, polycarboxylic acid, and one or more sorts of compounds with which it is chosen from the group to which it changes from those salts -- either -- the powder-coatings constituent of a publication.

[Claim 6] claims 1-4 to which the orthophosphoric acid monoester and/or orthophosphoric acid diester of the fatty alcohol used as a leafing-ized agent are guided from the fatty alcohol of carbon numbers 4-18 -- either -- the powder-coatings constituent of a publication.

[Claim 7] claims 1-6 whose additions of a leafing-ized agent are per [0.1] metal flake 100 weight section - 5 weight sections -- either -- the powder-coatings constituent of a publication.

[Claim 8] claims 1-7 by which the resin layer of per [0.1] metal flake 100 weight section - 50 weight sections is formed in the front face of a metal flake -- either -- the powder-coatings constituent of a publication.

[Claim 9] A leafing-ized agent to the paste-like constituent which serves as a metal flake from a part for a solvent liquefied or the constituent which carried out addition mixing in the state of the solution The process which coats a distributed media front face with a paste-like metal flake layer by distributing with distributed media, claims 2-8 which consist of a process which contacts thermosetting resin fine particles to the distributed media which coated this metal flake layer, and makes a metal flake imprint and adhere to a resin fine-particles front face, and a process which removes a part for a solvent by desiccation -- either -- the manufacture approach of the powder-coatings constituent a publication.

[Claim 10] a claim -- the paint film formation approach characterized by stiffening resin at the temperature of 150 degrees C or more after carrying out powder coating of the powder-coatings constituent of a publication to a base material one to 8 either

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Objects of the Invention] This invention relates to the powder-coatings constituent which gives the outstanding metallic feeling, its manufacture approach, and the paint film formation approach which used it.

[0002]

[Description of the Prior Art] As a low-pollution mold coating which does not use an organic solvent, need is increasing powder coatings to an automobile, an electrical home appliance, building materials, a toy, etc. However, when applying powder coatings to metallic coating finishing, since it becomes difficult to make metallic pigment arrange in parallel to a base material, a color tone becomes dark, and there is a problem that sufficient metallic feeling is not obtained.

[0003] Being conventionally developed as a fine-particles metallic paint has a method (BONDEDDO law) of making metallic pigment adhere to a powder-coatings resin front face by the approach (the melt blending method) of kneading metallic pigment enough with resin and a color pigment beforehand by scorification, a spray drying method, etc., the dryblend method for mixing and painting the metal flake powder-ized by drying with a resin particle, a brush polisher, etc. (For example, JP,51-137725,A, JP,57-35214,B, and U.S. Pat. No. 4,138,511 etc.)

In the melt blending method, it is easy to produce deformation of metallic pigment at the grain-refining process of the powder coatings by the kneading process or grinding, and a not much good appearance is not acquired. Furthermore, metallic pigment is ground, an activity front face is exposed, and there is also a problem that danger, such as ignition and dust explosion, becomes high.

[0004] Although it is hard to produce deformation of metallic pigment and the dryblend method is advantageous in [method / melt blending] design, it is difficult to carry out orientation of the aluminum flake in parallel with a base material too, and sufficient metallic feeling is not obtained. Since the electrification nature of resin fine particles and a metal flake furthermore differs, it is difficult for there to be a problem that the preparation presentation of powder coatings differs from the presentation of the painted paint film, and to carry out the reuse of the collected powder coatings.

[0005] By the BONDEDDO method, since metallic pigment is made to adhere on the surface of resin, the rate of metal flake installation to the inside of a paint film is stable, and there is a merit that the reuse of the powder coatings collected without adhering to a base material can be carried out. Moreover, although it is said that a metallic feeling is easy to be obtained since orientation of the metallic pigment can be carried out along a resin front face, a resin front face is not necessarily located in a line in parallel with a base material in fact, and sufficient metallic feeling is not obtained.

[Problem(s) to be Solved by the Invention] The main purposes of this invention tend to offer the powder-coatings constituent with which the problem of appearances, such as a metallic feeling which was the fault of metallic powder coatings conventionally, brightness, and paint film surface gloss, is solved, and it is satisfied of the fundamental requirements as powder coatings, such as a rate of metal flake installation, paint workability, recovery effectiveness, and chemical resistance.

[0006]

[Detailed Description of the Invention] The description of this invention is that it uses it, mixing with thermosetting resin fine particles in the dryblend method, or making the metal flake which made the leafing-ized agent stick to a front face adhere on the surface of thermosetting resin in the BONDEDDO method. Although dispersants, such as polycarboxylic acid of stearic acid, a myristic acid, a palmitic acid, a lauric acid, etc., such as saturated fatty acid of carbon numbers 12-18 and "Solsperse" (product name: AVECIA), and a salt of those, are illustrated as a leafing-ized agent, especially a desirable thing is the orthophosphoric acid monoester and/or the orthophosphoric acid diester, or those mixture of fatty alcohol. By using such a metallic powder-coatings constituent, the paint film which has the outstanding metallic feeling or the outstanding brightness can be obtained. moreover, BONDEDDO -- since the rate of metal flake installation to the inside of a paint film is stable when it is used as law and the reuse of a coating also becomes possible, it is advantageous also in respect of paint cost or an environmental cure. Furthermore, the problem of the paint film exfoliation which originated in using phosphoric ester conventionally and had become a problem is also solvable by applying this to powder coating.

[0007] It is desirable for the following resin to be illustrated as thermosetting resin and to use at least one sort in these thermosetting resin. The curing agent, the fluid regulator, the dispersant, etc. may be added by these resin if needed.

[0008] 5-100 micrometers of mean particle diameter of acrylic resin, polyester resin, polyurethane resin, and epoxy resin heat-curing resin fine particles are 15-60 micrometers more preferably. In less than 5 micrometers, the danger of the dust explosion at the time of manufacture or powder coating becomes high, and, in the case of the BONDEDDO method, mean particle diameter becomes difficult [making a metal flake adhere to a front face]. When mean particle diameter exceeds 100 micrometers, the smooth nature of the front face of a powder coating paint film is checked, and a good appearance is not acquired.

[0009] The orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol have the following structures, and are orthophosphoric acid diester of the orthophosphoric acid monoester: $R-O-PO(OH)_2$ fatty alcohol of fatty alcohol. : $(R-O)_2PO(OH)$

It is desirable for the following compounds to specifically be illustrated and to use at least one sort among these.

[0010] Stearyl acid phosphate, milli still acid phosphate, palmityl acid phosphate, lauryl acid phosphate, n-DESHIRU acid phosphate, 2 ethylhexyl acid phosphate, oleyl acid phosphate, hexyl acid phosphate, butyl acid phosphate.

[0011] Although these compounds are marketed as mixture of the orthophosphoric acid monoester of fatty alcohol, and orthophosphoric acid diester, they may usually mix and use that from which you may use it as the orthophosphoric acid monoester simple substance or orthophosphoric acid diester simple substance of fatty alcohol, and a carbon number differs.

[0012] Although it is indicated by JP,58-168663,A and is effective in improving the chemical resistance of a metal pigment etc., if such a metal flake is used for the usual coating, paint film physical properties will fall remarkably, and since there is a problem that the cohesive failure of a paint film arises easily by a tape peel test etc., addition to such a metal flake of a compound is hardly put in practical use. Moreover, there was also no chemical resistance of the metal flake obtained at sufficient thing.

[0013] In this invention, since the metal flake which added the orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol was applied to the powder coatings which carry out baking finish at an elevated temperature, the fall of paint film physical properties could be suppressed and it became possible to offer the paint film which is equal to practical use. Moreover, after covering resin beforehand on metal flakes, paint film physical properties can be further raised by adding the orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol. Furthermore, by adding the orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol on metal flakes showed that there was effectiveness also in a metallic feeling improvement of the powder coating paint film which is another purpose which was not known conventionally.

[0014] As for the carbon number of the fatty alcohol used as the raw material of the orthophosphoric

acid monoester of fatty alcohol, and/or orthophosphoric acid diester, it is desirable that it is 4-18. When a carbon number is three or less, sufficient color tone improvement effect is not acquired, but when a carbon number is 19 or more, the physical properties of a paint film fall, and problems, such as cohesive failure of a paint film, arise.

[0015] As for the addition of the orthophosphoric acid monoester of fatty alcohol to a metal flake, and/or orthophosphoric acid diester, it is desirable that they are per [0.1] metal flake 100 weight section - 5 weight sections, and it is 0.2 - 3 weight section more preferably. When an addition is under the 0.1 weight section, there is a possibility that sufficient color tone improvement effect may not be acquired, and when exceeding 5 weight sections, there is a possibility that the physical properties of a paint film may fall and problems, such as exfoliation of a paint film, may arise.

[0016] As a metal flake, bronze, stainless steel, etc. which are aluminum, copper, zinc, iron, chromium, titanium, nickel, or these alloys are illustrated. especially -- an aluminium flake -- weatherability, economical efficiency, and a paint film -- it is desirable in respect of description etc. The mean particle diameter of the metal flake for acquiring a good paint film appearance has more preferably 1-100-micrometer 0.01-5-micrometer 0.02-2 micrometers more preferably suitable for about 3-50 micrometers and average thickness. Matter other than the orthophosphoric acid monoester of fatty alcohol and/or orthophosphoric acid diester (oleic acid, stearic acid, etc.), for example, fatty acids, fatty amine, a fatty-acid amide, fatty alcohol, an ester compound, etc. may be sticking to the front face of a metal flake. Moreover, the coloring metal flake which formed other color pigment layers, interference film, etc. in the metal flake front face, and was colored it may be used.

[0017] It is desirable that the resin layer is formed in a metal flake front face. In this case, the resin layer of a surface of metal will be adsorbed in orthophosphoric acid monoester and/or orthophosphoric acid diester. By forming a resin layer in a metal flake front face, the chemical resistance of a paint film and the paint film physical properties which the addition effectiveness of the orthophosphoric acid monoester of fatty alcohol and/or orthophosphoric acid diester becomes large, and were painted upwards using this can be raised. The method of carrying out the polymerization of the monomer and depositing a polymer on a metal flake front face is desirable by adding polymerization initiators, such as azobisisobutyronitril and a benzoyl peroxide, adding a monomer as shown in the slurry which distributed the metal flake in the organic solvent as an approach of forming a resin layer below, and heating in an inert gas ambient atmosphere. However, it is not limited to this approach. As a monomer, it is illustrated below and at least one sort can use it suitably among these.

[0018] An acrylic acid, a methacrylic acid, a methyl methacrylate, (Example of a polymerization nature monomer) Butyl acrylate, 2-ethylhexyl acrylate, acrylic-acid lauryl, Acrylic-acid stearyl, acrylic-acid cyclohexyl, acrylic-acid 2-hydroxyethyl, Acrylic-acid 2-hydroxy butyl, acrylic-acid 2-methoxy ethyl, An acrylic-acid 2-diethylaminoethyl, methacrylic-acid butyl, methacrylic-acid octyl, 1,4-butanediol diacrylate, 1,6-hexanediol diacrylate, 1, 9 nonane diol diacrylate, neopentyl glycol diacrylate, Tripropylene glycol diacrylate, tetraethylene glycol diacrylate, Trimethylolpropane triacrylate, tetramethylolmethane tetraacrylate, A pentaerythritol thoria chestnut rate, tris acryloxy ethyl phosphate, Ditrimehylolpropanetetraacrylate, styrene, alpha methyl styrene, Vinyltoluene, a divinylbenzene, acrylic nitril, methacrylic nitril, Vinyl acetate, propionic-acid vinyl, a maleic acid, a crotonic acid, an itaconic acid, polybutadiene, the linseed oil, soybean oil, epoxidized soybean oil, epoxidation polybutadiene, cyclohexene vinyl mono-oxide, divinylbenzene mono-oxide.

[0019] The amount of the resin layer formed has per [0.1] metal flake 100 weight section - 50 desirable weight sections, and 0.5 - 40 weight section is more preferably suitable for it. The case of under the 0.1 weight section has a possibility that problems, like the color tone improvement effect by acid phosphate becomes insufficient [little chemical resistance of a paint film] may arise. When exceeding 50 weight sections, the metallic feeling or brightness of a powder coating paint film falls, and there is a possibility that the smooth nature on the front face of a paint film may also worsen.

[0020] It mixes with thermosetting resin fine particles, or the amount of the metal flake made to adhere to thermosetting resin fine particles has per [1] thermosetting resin fine-particles 100 weight section - 20 desirable weight sections, and it is 2 - 10 weight section more preferably. In order for there to be a

possibility that metallic feeling with the amount of a metal flake sufficient in under 1 weight section or brightness may not be obtained and to conceal a base material, the need of enlarging film thickness comes out. When exceeding 20 weight sections, the gloss of a paint film is lost and there is a possibility that an appearance may also worsen.

[0021] Although there is especially no limitation in the approach of making the metal flake to which the orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol were made sticking adhering to a heat-curing resin fine-particles front face when using it as a BONDED method, it sets in distributed equipment. After making the paste-like metal flake which contains an organic solvent in distributed media cover, the process which make resin fine particles contact, and a metal flake is made to imprint and adhere to resin fine particles, is finally dried, and removes an organic solvent component is desirable. Since the organic solvent contained in a paste-like metal flake makes a resin front face dissolve or swell at this time, it will be in the condition that a metal flake tends to adhere to a resin front face, positive adhesion is attained, and the configuration of a particle can be further made roundish [wore].

[0022] Although especially the organic solvent contained in a metal flake is not limited, a hydrocarbon system solvent, an ester solvent, ketones, an alcohols solvent, a glycol ether system solvent, etc. are used suitably. Moreover, water may be used when resin is water solubility. The amount of organic solvents contained in a paste-like metal flake is usually 5 - 50% more preferably 0.5 to 90%. If there are too few amounts of organic solvents, it will become difficult to make a metal flake adhere to a resin particle front face, if many [too], resin particles will fix and a big lump will be made.

[0023] As distributed media to be used, the ball of the quality of the material of steel with a diameter of about 0.5-10mm, an alumina, a zirconia, glass, etc. is desirable.

[0024] Although not limited especially about the equipment to be used, the dryer which has distributed devices, such as rolling mold dryers, such as a ball mill, a vibration mill, and a medium stirrer mill, an oscillatory type dryer, and a churning mold dryer, is illustrated. Especially the equipment as for which the distribution and the vacuum drying by distributed media are made to coincidence is desirable. As desiccation conditions, the equipment internal pressure of 30 or less Torr and the temperature of about 30-70 degrees C are desirable. When a pressure is high, temperature is too low and temperature is [drying efficiency is bad and] too high, there is a possibility that resin particles may carry out melting fixing and a big lump may be made.

[0025] The method of application of the powder-coatings constituent by this invention is painted to the base material which adjusted the front face by blasting processing, chemical conversion, etc. beforehand by the corona discharge method, the frictional electrification method, and the other powder coating approaches, it heats at 170-230 degrees C preferably, and the temperature of 150 degrees C or more and the approach of stiffening for 5 - 30 minutes preferably are suitable for it to it 1 minute or more. When whenever [stoving temperature] is low, problems, such as exfoliation of a paint film, are produced. As a base material, a griddle, a mild steel plate, an aluminum plate, and other metal plates are suitable.

[0026]

[Example] (Example 1) A commercial non leafing aluminium flake (Toyo Aluminium [K.K.] 7640NS nonvolatile matter: 65%) To 1l. of slurries which consider 15 micrometers and average thickness:0.4micro as a part for a metal, and contain it 100g, Mean particle diameter : 2.5g of methyl methacrylates, 1,6-hexanediol diacrylate 2.5g, styrene 2.5g, 2.5g (10 weight sections monomer sum total : to the aluminium flake 100 weight section) of glycidyl methacrylates is added. By heating at 80 degrees C in nitrogen, agitating, adding azobisisobutyronitril 0.5g as a polymerization initiator, and making it react for 12 hours, the polymerization of the monomer was carried out and the aluminium flake front face was deposited. Solid liquid separation of the slurry after processing was carried out, and it considered as the shape of a paste of 80% of solid content.

[0027] To obtained paste-like resin coat metal flake 100g, stearyl acid phosphate 0.8g was dissolved in mineral spirit 10g, and it added and mixed. The coating layer of a paste-like resin coat aluminium flake was formed in the alumina-balls front face by making a cylindrical cup with a diameter [of 12cm], and a die length of 20cm insert and rotate 11g [of obtained paste-like constituents], and with a diameter of

3mm alumina-balls 1000g. Then, the aluminium flake was made to imprint and adhere to a thermosetting resin fine-particles front face by adding 100g (KUBOKO PAINT Co., Ltd. TEODEYURU PE 785-900) of thermosetting commercial polyester resin fine particles for powder coating to this cylindrical cup, and making it rotate a cylindrical cup further. After the screen finally separated a ball and resin, hot air drying was carried out at 40 degrees C for 2 hours, and the metallic powder-coatings constituent was obtained. In addition, in the above, when it replaced with stearyl ASHISHIDO phosphate and polycarboxylic acid ("Solsperse" #9000) was used, the powder coatings of the engine performance almost equivalent to the case (example 27) where stearic acid is used were obtained.

[0028] (Example 2) It considered as the same process as an example 1 until it obtained the paste-like resin coat metal flake of 80% of solid content. After dissolving steer RIRURU acid phosphate 0.8g in mineral spirit 10g and adding and mixing to obtained paste-like resin coat metal flake 100g, it powder-ized by carrying out a vacuum drying by the evaporator.

[0029] Aluminium flake 8g and 100g (KUBOKO PAINT Co., Ltd. TEODEYURU PE 785-900) of thermosetting polyester resin fine particles for powder coating powder-ized by the above were mixed by the V shaped rotary mixer, and the metallic powder-coatings constituent was obtained.

[0030] (Example 3) To non leafing aluminium flake (Toyo Aluminium K.K. 7640NS) 100g, stearyl acid phosphate 0.7g was dissolved in mineral spirit 5g, and it added and mixed. The coating layer of a paste-like aluminium flake was formed in the alumina-balls front face by making a cylindrical cup with a diameter [of 12cm], and a die length of 20cm insert and rotate 12g [of obtained paste-like constituents], and with a diameter of 3mm alumina-balls 1000g (100rpm). Then, the aluminium flake was made to imprint and adhere to a thermosetting resin fine-particles front face by adding 100g (KUBOKO PAINT Co., Ltd. TEODEYURU PE 785-900) of thermosetting polyester resin fine particles for powder coating to this cylindrical cup, and making it rotate a cylindrical cup further. After the screen finally separated a ball and resin, hot air drying was carried out at 40 degrees C for 2 hours, and the metallic powder-coatings constituent was obtained.

[0031] (Example 4) To non leafing aluminium flake (Toyo Aluminium K.K. 7640NS) 100g, stearyl acid phosphate 0.7g was dissolved in mineral spirit 5g, and it added and mixed. By carrying out the vacuum drying of the obtained paste-like constituent by the evaporator, aluminium flake 8g and 100g (KUBOKO PAINT Co., Ltd. TEODEYURU PE 785-900) of thermosetting polyester resin fine particles for powder coating powder-ized were mixed by the V shaped rotary mixer, and the metallic powder-coatings constituent was obtained.

[0032] (Examples 5-30, examples 1-12 of a comparison) except for having changed the class of metal pigment constituent, the class of thermosetting resin fine particles, and the amount, as shown in Table 1 -- BONDEDDO -- in law, the metallic powder-coatings constituent of examples 5-30 and the examples 1-12 of a comparison was produced like examples 2 and 4 in the dryblend method like examples 1 and 3. In Table 1, 0630M are a Lee fin GUARUMIPE [by Toyo Aluminium K.K.]-strike (70% mean particle diameter of 15 micrometers of nonvolatile matters, average thickness of 1.4 micrometers), and RE2600 is a coloring ARUMIPE [by Toyo Aluminium K.K.]-strike (mean particle diameter of 18 micrometers, average thickness of 1.7 micrometers).

[0033] (Test) Electrostatic powder coating (use model: MATSUO SANGYO Co., Ltd. MPSI-C mold) was carried out to three tin plates with the applied voltage of 90kV by having made into the base enamel the powder coatings obtained in examples 1-30 and the examples 1-12 of a comparison, and two sheets were able to be burned for 20 minutes at 180 degrees C. These are made into color card ** and color card **. The paint film thickness of color card [at this time] ** and ** was 50 micrometers.

[0034] Then, powder coating of the same clear resin fine particles as the resin used for the base enamel of this color card was further carried out to color card **, and it was able to be again burned for 20 minutes at 180 degrees C. The thickness of the top clear layer at this time was 40 micrometers.

[0035] It evaluated by measuring L value (it being called L15 value below) in 45 degrees of incident angles according the metallic feeling of obtained color card ** to a deflection colorimeter (X-Rite MA68 made from X-Rite), and 15 degrees of offset angles from specular reflection. (The one where L

value is larger is metallic feeling fitness.) Viewing estimated five steps of the appearances (gloss) and brightness of a paint film again. (2: Superiority, 4: Five : fitness, 3: usually inferior one: defect.) It is JIS about the adhesion of the top clear layer of obtained color card ** further. The squares tape method by K5400 estimated ten steps. As for the evaluating point at this time, according to JIS, 10 is good, there is no peeling, and, as for 65% or more of peeling, 0 generates and is poor.

[0036] Moreover, the chemical resistance of the paint film of color card ** was tested based on JIS K5400, and five-step evaluation was performed. (2: 5: -- superiority, 4:fitness, and 3: -- usually make inferior 1: into a defect.) test liquid was used [alkali resistance] as the sulfuric-acid solution 5% about 5% solution of sodium carbonates, and acid resistance, and both test time amount was made into 24 hours.

[0037] About one more sheet, before the adhering coating could be burned, it collected from the tin plate, and after heating and ashing at 800 degrees C among air, EDTA titrimetric analysis analyzed a part for the metal contained in it, and it carried out the quantum. By teaching a part for the analyzed metal (%), breaking by part for the metal under presentation (%), and being shown by the percentage, it considered as the rate of installation of a metal flake. The obtained result is shown in Table 2.

[0038] The microphotography of the cross section of color card ** obtained by drawing 1 in the example 1 is shown. It turns out that aluminum deflection-KU was distributed not only over the interior of a paint film but over the paint film front face from this drawing, and it has contributed to the outstanding brightness.

other test-methods: -- mean-particle-diameter: -- average thickness: by the laser diffraction type particle-size-distribution measuring method -- the observation (average of ten particles of arbitration) by the SEM observation photograph

[0039]

[Table 1]

表1 粉体塗料組成物の構成

	金属顔料組成物の種類				樹脂粉体の種類	Alフレーク対樹脂粉体(重量)	ボンデッド法(B) or ドライブレンド法(D)
	原料金属フレークの種類(品名)	樹脂コート量(重量% vs Alフレーク)	リフティング化剤の種類	リフティング化剤添加量(重量% vs Alフレーク)			
実施例1	7640NS	10	ステアリン酸ジブチルホスフェート	1.1	ポリエステル	7.2/100	B
実施例2	7640NS	10	ステアリン酸ジブチルホスフェート	1.1	ポリエステル	7.2/100	D
実施例3	7640NS	—	ステアリン酸ジブチルホスフェート	1.0	ポリエステル	8.0/100	B
実施例4	7640NS	—	ステアリン酸ジブチルホスフェート	1.0	ポリエステル	7.9/100	D
実施例5	7640NS	10	ラウリルアジテートホスフェート	1.1	ポリエステル	7.2/100	B
実施例6	7640NS	10	2-エチルヘキシルアジテートホスフェート	1.1	ポリエステル	7.2/100	B
実施例7	7640NS	—	ステアリン酸ジブチルホスフェート	3.0	ポリエステル	7.8/100	B
実施例8	7640NS	10	ブチルアジテートホスフェート	1.1	ポリエステル	7.2/100	B
実施例9	7640NS	10	エイコシルアジテートホスフェート	1.1	ポリエステル	7.2/100	B
実施例10	0630M	—	ラウリルアジテートホスフェート	1.0	アクリル	4.0/100	B
実施例11	RE2600	—	ラウリルアジテートホスフェート	1.0	ポリエステル	8.0/100	B
実施例12	7640NS	10	ラウリルアジテートホスフェート	3.3	ポリエステル	7.1/100	B
実施例13	7640NS	10	ラウリルアジテートホスフェート	6.6	ポリエステル	7.0/100	B
実施例14	7640NS	10	ラウリルアジテートホスフェート	0.22	ポリエステル	7.3/100	B
実施例15	7640NS	10	ラウリルアジテートホスフェート	0.06	ポリエステル	7.3/100	B
実施例16	7640NS	0.2	ラウリルアジテートホスフェート	1.0	ポリエステル	8.0/100	B
実施例17	7640NS	0.05	ラウリルアジテートホスフェート	6.0	ポリエステル	7.7/100	B
実施例18	7640NS	30	ラウリルアジテートホスフェート	0.26	ポリエステル	6.1/100	B
実施例19	7640NS	60	ラウリルアジテートホスフェート	0.08	ポリエステル	5.0/100	B
実施例20	7640NS	10	ラウリルアジテートホスフェート	1.1	アクリル	7.2/100	B
実施例21	7640NS	10	ラウリルアジテートホスフェート	1.1	ポリウレタン	7.2/100	B
実施例22	7640NS	10	ラウリルアジテートホスフェート	1.1	ポリエステル	3.6/100	B
実施例23	0630M	—	ラウリルアジテートホスフェート	1.0	ポリエステル	0.5/100	B
実施例24	7640NS	10	ラウリルアジテートホスフェート	1.1	ポリエステル	13.5/100	B
実施例25	7640NS	10	ラウリルアジテートホスフェート	1.1	ポリエステル	22.6/100	B
実施例26	7640NS	10	2-エチルヘキシルアジテートホスフェート	1.1	アクリル	7.2/100	D
実施例27	7640NS	10	ステアリン酸	3.3	ポリエステル	7.1/100	D
実施例28	7640NS	10	ブチルアジテートホスフェート	1.1	ポリエステル	18.0/100	D
実施例29	RE2600	—	ラウリルアジテートホスフェート	1.0	ポリウレタン	7.9/100	D
実施例30	0630M	—	ラウリルアジテートホスフェート	1.0	ポリエステル	7.9/100	D
比較例1	7640NS	—	—	—	ポリエステル	8.0/100	B
比較例2	7640NS	—	—	—	ポリエステル	8.0/100	D
比較例3	7640NS	10	—	—	ポリエステル	8.0/100	B
比較例4	7640NS	10	—	—	ポリエステル	7.3/100	D
比較例5	7640NS	10	—	—	アクリル	8.0/100	B
比較例6	7640NS	10	—	—	アクリル	7.3/100	D
比較例7	7640NS	10	—	—	ポリウレタン	8.0/100	B
比較例8	7640NS	10	—	—	ポリウレタン	7.3/100	D
比較例9	RE2600	—	—	—	ポリエステル	8.0/100	B
比較例10	RE2600	—	—	—	ポリエステル	7.3/100	D
比較例11	0630M	—	—	—	アクリル	4/100	B
比較例12	0630M	—	—	—	アクリル	3.6/100	D

[0040]

[Table 2]

表2 粉体塗料の評価結果

	L15値	目視評価		密着性	塗膜耐薬品性		金属フー 導入手率 (%)
		外観	光輝感		耐アルカリ	耐酸性	
実施例1	179	5	5	10	5	5	100
実施例2	210	5	5	10	5	5	66
実施例3	145	5	3	10	3	4	98
実施例4	123	5	3	10	3	4	52
実施例5	177	5	5	10	5	5	99
実施例6	165	5	4	10	5	5	99
実施例7	170	5	5	8	3	4	97
実施例8	140	5	3	10	5	5	100
実施例9	175	5	5	8	5	5	100
実施例10	160	5	5	8	3	4	96
実施例11	97	5	5	10	5	5	100
実施例12	186	5	5	9	5	5	99
実施例13	185	5	5	6	5	5	100
実施例14	150	5	4	10	5	5	100
実施例15	145	5	3	10	5	5	99
実施例16	152	5	4	10	4	5	98
実施例17	144	5	3	9	3	4	97
実施例18	169	5	4	10	5	5	100
実施例19	152	5	3	10	5	5	100
実施例20	116	5	3	10	5	5	99
実施例21	181	5	5	10	5	5	98
実施例22	101	5	3	10	5	5	100
実施例23	137	5	3	10	5	5	99
実施例24	187	5	5	9	4	5	97
実施例25	184	5	5	8	3	5	98
実施例26	191	5	4	10	5	5	54
実施例27	175	4	4	8	5	5	48
実施例28	155	5	4	10	4	5	45
実施例29	105	5	5	10	5	5	74
実施例30	169	5	3	10	3	5	65
比較例1	105	5	2	8	2	5	90
比較例2	67	3	1	8	2	3	30
比較例3	112	5	3	10	5	5	99
比較例4	135	4	4	10	5	5	51
比較例5	75	5	2	10	5	5	100
比較例6	81	4	2	10	5	5	49
比較例7	72	5	2	10	5	5	99
比較例8	90	4	3	10	5	5	51
比較例9	65	5	2	10	5	5	99
比較例10	78	4	3	10	5	5	59
比較例11	103	5	3	6	2	3	91
比較例12	121	3	4	8	2	3	63

[0041] (Example 31) Electrostatic powder coating of the powder-coatings constituent obtained in the example 3 was carried out, and it was able to be burned for 20 minutes at 160 degrees C. Then, powder coating of the same clear resin fine particles as the resin used for this color card was carried out further, and it was able to be again burned for 20 minutes at 160 degrees C.

[0042] (Example 13 of a comparison) Electrostatic powder coating of the powder-coatings constituent obtained in the example 3 was carried out, and it was able to be burned for 20 minutes at 140 degrees C. Then, powder coating of the same clear resin fine particles as the resin used for this color card was carried out further, and it was able to be again burned for 20 minutes at 140 degrees C.

[0043] (Example 14 of a comparison) Electrostatic powder coating of the powder-coatings constituent obtained in the example 1 was carried out, and it was able to be burned for 20 minutes at 140 degrees C. Then, powder coating of the same clear resin fine particles as the resin used for this color card was carried out further, and it was able to be again burned for 20 minutes at 140 degrees C.

[0044] The results of the peel test by the squares tape method of the color card obtained in the example 31, the example 13 of a comparison, and the example 14 of a comparison were the evaluating points 8, 3, and 3, respectively.

[Translation done.]

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PRIOR ART

[Description of the Prior Art] As a low-pollution mold coating which does not use an organic solvent, need is increasing powder coatings to an automobile, an electrical home appliance, building materials, a toy, etc. However, when applying powder coatings to metallic coating finishing, since it becomes difficult to make metallic pigment arrange in parallel to a base material, a color tone becomes dark, and there is a problem that sufficient metallic feeling is not obtained.

[0003] Being conventionally developed as a fine-particles metallic paint has a method (BONDEDDO law) of making metallic pigment adhere to a powder-coatings resin front face by the approach (the melt blending method) of kneading metallic pigment enough with resin and a color pigment beforehand by scorification, a spray drying method, etc., the dryblend method for mixing and painting the metal flake powder-ized by drying with a resin particle, a brush polisher, etc. (For example, JP,51-137725,A, JP,57-35214,B, and U.S. Pat. No. 4,138,511 etc.)

In the melt blending method, it is easy to produce deformation of metallic pigment at the grain-refining process of the powder coatings by the kneading process or grinding, and a not much good appearance is not acquired. Furthermore, metallic pigment is ground, an activity front face is exposed, and there is also a problem that danger, such as ignition and dust explosion, becomes high.

[0004] Although it is hard to produce deformation of metallic pigment and the dryblend method is advantageous in [method / melt blending] design, it is difficult to carry out orientation of the aluminum flake in parallel with a base material too, and sufficient metallic feeling is not obtained. Since the electrification nature of resin fine particles and a metal flake furthermore differs, it is difficult for there to be a problem that the preparation presentation of powder coatings differs from the presentation of the painted paint film, and to carry out the reuse of the collected powder coatings.

[0005] By the BONDEDDO method, since metallic pigment is made to adhere on the surface of resin, the rate of metal flake installation to the inside of a paint film is stable, and there is a merit that the reuse of the powder coatings collected without adhering to a base material can be carried out. Moreover, although it is said that a metallic feeling is easy to be obtained since orientation of the metallic pigment can be carried out along a resin front face, a resin front face is not necessarily located in a line in parallel with a base material in fact, and sufficient metallic feeling is not obtained.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] The main purposes of this invention tend to offer the powder-coatings constituent with which the problem of appearances, such as a metallic feeling which was the fault of metallic powder coatings conventionally, brightness, and paint film surface gloss, is solved, and it is satisfied of the fundamental requirements as powder coatings, such as a rate of metal flake installation, paint workability, recovery effectiveness, and chemical resistance.

[0006]

[Detailed Description of the Invention] The description of this invention is that it uses it, mixing with thermosetting resin fine particles in the dryblend method, or making the metal flake which made the leafing-ized agent stick to a front face adhere on the surface of thermosetting resin in the BONDEDDO method. Although dispersants, such as polycarboxylic acid of stearic acid, a myristic acid, a palmitic acid, a lauric acid, etc., such as saturated fatty acid of carbon numbers 12-18 and "Solsperse" (product name: AVECIA), and a salt of those, are illustrated as a leafing-ized agent, especially a desirable thing is the orthophosphoric acid monoester and/or the orthophosphoric acid diester, or those mixture of fatty alcohol. By using such a metallic powder-coatings constituent, the paint film which has the outstanding metallic feeling or the outstanding brightness can be obtained. moreover, BONDEDDO -- since the rate of metal flake installation to the inside of a paint film is stable when it is used as law and the reuse of a coating also becomes possible, it is advantageous also in respect of paint cost or an environmental cure. Furthermore, the problem of the paint film exfoliation which originated in using phosphoric ester conventionally and had become a problem is also solvable by applying this to powder coating.

[0007] It is desirable for the following resin to be illustrated as thermosetting resin and to use at least one sort in these thermosetting resin. The curing agent, the fluid regulator, the dispersant, etc. may be added by these resin if needed.

[0008] 5-100 micrometers of mean particle diameter of acrylic resin, polyester resin, polyurethane resin, and epoxy resin heat-curing resin fine particles are 15-60 micrometers more preferably. In less than 5 micrometers, the danger of the dust explosion at the time of manufacture or powder coating becomes high, and, in the case of the BONDEDDO method, mean particle diameter becomes difficult [making a metal flake adhere to a front face]. When mean particle diameter exceeds 100 micrometers, the smooth nature of the front face of a powder coating paint film is checked, and a good appearance is not acquired.

[0009] The orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol have the following structures, and are orthophosphoric acid diester of the orthophosphoric acid monoester: $R-O-PO(OH)_2$ fatty alcohol of fatty alcohol. : $(R-O)_2PO(OH)$

It is desirable for the following compounds to specifically be illustrated and to use at least one sort among these.

[0010] Stearyl acid phosphate, myristyl acid phosphate, palmityl acid phosphate, lauryl acid phosphate, n-DESHIRU acid phosphate, 2 ethylhexyl acid phosphate, oleyl acid phosphate, hexyl acid phosphate, butyl acid phosphate.

[0011] Although these compounds are marketed as mixture of the orthophosphoric acid monoester of

fatty alcohol, and orthophosphoric acid diester, they may usually mix and use that from which you may use it as the orthophosphoric acid monoester simple substance or orthophosphoric acid diester simple substance of fatty alcohol, and a carbon number differs.

[0012] Although it is indicated by JP,58-168663,A and is effective in improving the chemical resistance of a metal pigment etc., if such a metal flake is used for the usual coating, paint film physical properties will fall remarkably, and since there is a problem that the cohesive failure of a paint film arises easily by a tape peel test etc., addition to such a metal flake of a compound is hardly put in practical use.

Moreover, there was also no chemical resistance of the metal flake obtained at sufficient thing.

[0013] In this invention, since the metal flake which added the orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol was applied to the powder coatings which carry out baking finish at an elevated temperature, the fall of paint film physical properties could be suppressed and it became possible to offer the paint film which is equal to practical use. Moreover, after covering resin beforehand on metal flakes, paint film physical properties can be further raised by adding the orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol. Furthermore, by adding the orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol on metal flakes showed that there was effectiveness also in a metallic feeling improvement of the powder coating paint film which is another purpose which was not known conventionally.

[0014] As for the carbon number of the fatty alcohol used as the raw material of the orthophosphoric acid monoester of fatty alcohol, and/or orthophosphoric acid diester, it is desirable that it is 4-18. When a carbon number is three or less, sufficient color tone improvement effect is not acquired, but when a carbon number is 19 or more, the physical properties of a paint film fall, and problems, such as cohesive failure of a paint film, arise.

[0015] As for the addition of the orthophosphoric acid monoester of fatty alcohol to a metal flake, and/or orthophosphoric acid diester, it is desirable that they are per [0.1] metal flake 100 weight section - 5 weight sections, and it is 0.2 - 3 weight section more preferably. When an addition is under the 0.1 weight section, there is a possibility that sufficient color tone improvement effect may not be acquired, and when exceeding 5 weight sections, there is a possibility that the physical properties of a paint film may fall and problems, such as exfoliation of a paint film, may arise.

[0016] As a metal flake, bronze, stainless steel, etc. which are aluminum, copper, zinc, iron, chromium, titanium, nickel, or these alloys are illustrated. especially -- an aluminium flake -- weatherability, economical efficiency, and a paint film -- it is desirable in respect of description etc. The mean particle diameter of the metal flake for acquiring a good paint film appearance has more preferably 1-100-micrometer 0.01-5-micrometer 0.02-2 micrometers more preferably suitable for about 3-50 micrometers and average thickness. Matter other than the orthophosphoric acid monoester of fatty alcohol and/or orthophosphoric acid diester (oleic acid, stearic acid, etc.), for example, fatty acids, fatty amine, a fatty-acid amide, fatty alcohol, an ester compound, etc. may be sticking to the front face of a metal flake. Moreover, the coloring metal flake which formed other color pigment layers, interference film, etc. in the metal flake front face, and was colored it may be used.

[0017] It is desirable that the resin layer is formed in a metal flake front face. In this case, the resin layer of a surface of metal will be adsorbed in orthophosphoric acid monoester and/or orthophosphoric acid diester. By forming a resin layer in a metal flake front face, the chemical resistance of a paint film and the paint film physical properties which the addition effectiveness of the orthophosphoric acid monoester of fatty alcohol and/or orthophosphoric acid diester becomes large, and were painted upwards using this can be raised. The method of carrying out the polymerization of the monomer and depositing a polymer on a metal flake front face is desirable by adding polymerization initiators, such as azobisisobutyronitril and a benzoyl peroxide, adding a monomer as shown in the slurry which distributed the metal flake in the organic solvent as an approach of forming a resin layer below, and heating in an inert gas ambient atmosphere. However, it is not limited to this approach. As a monomer, it is illustrated below and at least one sort can use it suitably among these.

[0018] An acrylic acid, a methacrylic acid, a methyl methacrylate, (Example of a polymerization nature monomer) Butyl acrylate, 2-ethylhexyl acrylate, acrylic-acid lauryl, Acrylic-acid stearyl, acrylic-acid

cyclohexyl, acrylic-acid 2-hydroxyethyl, Acrylic-acid 2-hydroxy butyl, acrylic-acid 2-methoxy ethyl, An acrylic-acid 2-diethylaminoethyl, methacrylic-acid butyl, methacrylic-acid octyl, 1,4-butanediol diacrylate, 1,6-hexanediol diacrylate, 1, 9 nonane diol diacrylate, neopentyl glycol diacrylate, Tripropylene glycol diacrylate, tetraethylene glycol diacrylate, Trimethylolpropane triacrylate, tetramethylolmethane tetraacrylate, A pentaerythritol thoria chestnut rate, tris acryloxy ethyl phosphate, Ditrimehylolpropanetetraacrylate, styrene, alpha methyl styrene, Vinyltoluene, a divinylbenzene, acrylic nitril, methacrylic nitril, Vinyl acetate, propionic-acid vinyl, a maleic acid, a crotonic acid, an itaconic acid, polybutadiene, the linseed oil, soybean oil, epoxidized soybean oil, epoxidation polybutadiene, cyclohexene vinyl mono-oxide, divinylbenzene mono-oxide.

[0019] The amount of the resin layer formed has per [0.1] metal flake 100 weight section - 50 desirable weight sections, and 0.5 - 40 weight section is more preferably suitable for it. The case of under the 0.1 weight section has a possibility that problems, like the color tone improvement effect by acid phosphate becomes insufficient [little chemical resistance of a paint film] may arise. When exceeding 50 weight sections, the metallic feeling or brightness of a powder coating paint film falls, and there is a possibility that the smooth nature on the front face of a paint film may also worsen.

[0020] It mixes with thermosetting resin fine particles, or the amount of the metal flake made to adhere to thermosetting resin fine particles has per [1] thermosetting resin fine-particles 100 weight section - 20 desirable weight sections, and it is 2 - 10 weight section more preferably. In order for there to be a possibility that metallic feeling with the amount of a metal flake sufficient in under 1 weight section or brightness may not be obtained and to conceal a base material, the need of enlarging film thickness comes out. When exceeding 20 weight sections, the gloss of a paint film is lost and there is a possibility that an appearance may also worsen.

[0021] Although there is especially no limitation in the approach of making the metal flake to which the orthophosphoric acid monoester and/or orthophosphoric acid diester of fatty alcohol were made sticking adhering to a heat-curing resin fine-particles front face when using it as a BONDEDDO method, it sets in distributed equipment. After making the paste-like metal flake which contains an organic solvent in distributed media cover, the process which make resin fine particles contact, and a metal flake is made to imprint and adhere to resin fine particles, is finally dried, and removes an organic solvent component is desirable. Since the organic solvent contained in a paste-like metal flake makes a resin front face dissolve or swell at this time, it will be in the condition that a metal flake tends to adhere to a resin front face, positive adhesion is attained, and the configuration of a particle can be further made roundish [wore].

[0022] Although especially the organic solvent contained in a metal flake is not limited, a hydrocarbon system solvent, an ester solvent, ketones, an alcohols solvent, a glycol ether system solvent, etc. are used suitably. Moreover, water may be used when resin is water solubility. The amount of organic solvents contained in a paste-like metal flake is usually 5 - 50% more preferably 0.5 to 90%. If there are too few amounts of organic solvents, it will become difficult to make a metal flake adhere to a resin particle front face, if many [too], resin particles will fix and a big lump will be made.

[0023] As distributed media to be used, the ball of the quality of the material of steel with a diameter of about 0.5-10mm, an alumina, a zirconia, glass, etc. is desirable.

[0024] Although not limited especially about the equipment to be used, the dryer which has distributed devices, such as rolling mold dryers, such as a ball mill, a vibration mill, and a medium stirrer mill, an oscillatory type dryer, and a churning mold dryer, is illustrated. Especially the equipment as for which the distribution and the vacuum drying by distributed media are made to coincidence is desirable. As desiccation conditions, the equipment internal pressure of 30 or less Torr and the temperature of about 30-70 degrees C are desirable. When a pressure is high, temperature is too low and temperature is [drying efficiency is bad and] too high, there is a possibility that resin particles may carry out melting fixing and a big lump may be made.

[0025] The method of application of the powder-coatings constituent by this invention is painted to the base material which adjusted the front face by blasting processing, chemical conversion, etc. beforehand by the corona discharge method, the frictional electrification method, and the other powder coating

approaches, it heats at 170-230 degrees C preferably, and the temperature of 150 degrees C or more and the approach of stiffening for 5 - 30 minutes preferably are suitable for it to it 1 minute or more. When whenever [stoving temperature] is low, problems, such as exfoliation of a paint film, are produced. As a base material, a griddle, a mild steel plate, an aluminum plate, and other metal plates are suitable.

[Translation done.]